

Sequential Batch Leach Test
U.S. Army Corps of Engineers

Lab Manager Approval _____ QA/QC Manager Approval _____

1.0 PURPOSE AND SCOPE

This procedure describes methods, materials, equipment, and special conditions required to determine guidance on the conduct of the anaerobic sequential batch leach testing.

2.0 EQUIPMENT

- 2.1 450-mL stainless steel centrifuge tubes for organics
- 2.2 250-mL polycarbonate centrifuge tubes for metals
- 2.3 Balance with precision to ± 0.01 g for samples having mass of 200g, and a precision of 0.1g for samples over 200g.
- 2.4 Glove box of sufficient size to contain centrifuge bottles, sediment, and balance.
- 2.5 Nitrogen
- 2.6 Vacuum source
- 2.7 Mechanical mixer
- 2.8 Stainless steel spatula
- 2.9 Paper towels
- 2.10 Glass fiber filter, 1 μ m, 47mm diameter, binder free
- 2.11 Glass fiber prefilter, 4 μ m, 47mm diameter, binder free
- 2.12 Cellulos acetate filters, 0.45 μ m, 47mm diameter
- 2.13 Filtration manifolds for organics and metals
- 2.14 High capacity tumbler
- 2.15 Muffle furnace
- 2.16 Oxygen meter
- 2.17 1-L amber glass sample bottles for organic contaminants
- 2.18 250-mL plastic sample bottles for metals
- 2.19 Contaminated sediment

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3.0 REAGENT

- 3.1 Deionized water
- 3.2 Concentrated HCl
- 3.3 Concentrated Ultrex HNO₃

4.0 PROCEDURE

- 4.1 For organic contaminant leaching, use clean stainless steel centrifuge tubes, stainless steel spatulas, and glass filtration apparatus. Combust glass fiber filter and prefilter at 400°C for 15 minutes.
- 4.2 For metal contaminant leaching, use clean polycarbonate centrifuge tubes, stainless steel spatulas, and polycarbonate filtration apparatus.
- 4.3 Prepare forms and labels. Conduct percent solids determination on mixed sediment sample and calculate solids and water content and required weights of water and sediment to achieve a water-to-solids ratio of 4:1 (weight of pore water + weight of DI water/dry weight of sediment).
- 4.4 Seal the glove box, purge with nitrogen until oxygen meter registers less than 1 percent. Ensure that a slight overpressure of nitrogen exists inside the glove box. This can be determined by observation of a slight expansion of the rubber gloves attached to the glove box.
- 4.5 Add all necessary equipment to the glove box through the airlock. Continue to purge box with nitrogen to remove any residual oxygen.
- 4.6 In the glove box, homogenize the sediment to ensure uniformity. Place a centrifuge bottle with cap on the balance and record the weight. Tare the centrifuge bottle and cap and load with sediment to the desired weight. Record the weight of the sediment added. Tare the centrifuge bottle, cap, and added sediment and add DI water to bring the final water-sediment ratio to 4:1. Wipe sediment from any surface that contacts the O-ring of the leakproof top. Record the weight of DI water, then zero the balance and record the weight of bottle cap, sediment, and leach water. Bottles should be loaded

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- in pairs of bottles balance to within 2g. For organic contaminants, multiple bottles may be required to obtain sufficient leachate (1 L) for chemical analysis.
- 4.7 Ensure that all centrifuge bottles are sealed, then remove the bottles from the glove box and transfer them to a tumbler. Tumble the samples for 24 hours at a rate of 40 rpm. Record the time tumbling starts and stops.
- 4.8 Remove the centrifuge bottles from the tumbler and place paired bottles opposite one another in a refrigerated centrifuge. Centrifuge stainless steel tubes for organic contaminant analysis at 6,500 x g for 30 minutes. Note: Stainless steel centrifuge tubes are heavy, limiting the speed of centrifugation. Leachates for metals are centrifuged at 9,000 x g.
- 4.9 Assemble the decontaminated filtration apparatus. For organic contaminants, the 4- μ m prefilter is placed over the 1- μ m glass fiber filter. Filter the samples; maintaining a nitrogen atmosphere over the samples while filtration is ongoing. Acidify leachate for organic analysis with 1 mL of concentrated HCl per liter of leachate to prevent iron precipitation and organic scavenging, then transfer sample to a precleaned, 1L amber glass bottle. Bottles for analysis of organic contaminants should be filled to the top. For metals, much the same procedure is followed. Filter the sample through a 0.45- μ m filter and acidify with 1 mL of concentrated Ultrex nitric acid per liter of leachate. Transfer leachate samples to plastic bottles for storage and analysis.
- 4.10 In the deoxygenated glove box, record the weight of the centrifuge bottle with lid and sediment after filtering. Repeat with remaining samples.
- 4.11 Add DI water to the centrifuge tubes to bring them back to the same water-to-solids ratio of 4:1. Record the weight of bottle with lid, DI water, and sediment. Repeat with remaining samples.
- 4.12 Tumble samples and centrifuge as described in 4.7 thru 4.9. Repeat a minimum of four times.
- 4.13 Using DI water, prepare and run a procedure blank according to the procedure described above for one cycle.
- 4.14 Clean workstation when work is finished. Initial and date data sheet.

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5.0 CALCULATIONS

TBD

6.0 SAFETY

- 6.1 Lab wear including a lab coat, safety goggles, and gloves should be worn at all times.
- 6.2 Keep workstation clean at all times. Wipe any spills to avoid safety hazards.
- 6.3 Be aware of all procedures when handling hazardous samples.

7.0 REPORT

TBD